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4,4′,6,6′-Tetrachloro-2,2′-[(1*E*,1′*E*)-propane-1,3-diylbis(nitrilomethanylylidene)]diphenol

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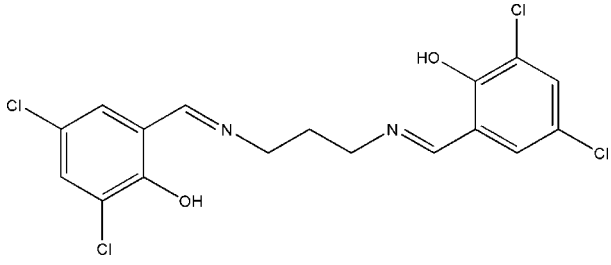
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.068; data-to-parameter ratio = 17.0.

The title compound, $C_{17}H_{14}Cl_4N_2O_2$, is generated by crystallographic twofold symmetry. The two benzene rings are inclined to one another by 80.17 (10)°. There are two intramolecular O—H···N hydrogen bonds, which make S(6) ring motifs. In the crystal, molecules are linked by C—H···O and weak C—H···Cl interactions, forming a three-dimensional network.

Related literature

For standard bond lengths, see: Allen *et al.*, (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related Schiff base ligands, see: Kargar *et al.* (2011); Kia *et al.* (2010).



Experimental

Crystal data

$C_{17}H_{14}Cl_4N_2O_2$ M_r = 420.10
Orthorhombic, *Fdd*2
a = 24.9797 (14) Å
b = 31.666 (3) Å
c = 4.4495 (2) Å
V = 3519.6 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.69 mm^{-1}
T = 291 K
0.26 × 0.23 × 0.18 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.842, T_{max} = 0.886
7926 measured reflections
1960 independent reflections
1634 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.04
1960 reflections
115 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 842 Friedel pairs
Flack parameter: 0.08 (7)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.84	2.574 (2)	147
C5—H2···O1^{i}	0.93	2.43	3.336 (2)	166
C8—H5B···Cl1^{ii}	0.97	2.89	3.851 (2)	169

Symmetry codes: (i) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{1}{4}$; (ii) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{5}{4}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2464).

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supplementary materials

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Comment

In continuation of our work on the crystal structure analyses of Schiff base ligands (Kargar *et al.*, (2011); Kia *et al.*, (2010), we synthesized the title compound and report herein on its crystal structure.

The title compound, Fig. 1, a potential tetradentate Schiff base ligand, possesses two-fold rotation symmetry, atom C9 is located on the 2-fold axis. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges. The two symmetry related benzene rings are inclined to one another by 80.17 (10) °. There are two intramolecular O—H···N hydrogen bonds which make *S(6)* ring motifs (Table 1; Bernstein *et al.*, 1995).

In the crystal, molecules are linked by C—H···O and weak C—H···Cl interactions to form a three-dimensional network (Table 1 and Fig. 2).

Experimental

The title compound was synthesized by adding 3,5-dichlorosalicylaldehyde (2 mmol) to a solution of propylenediamine (1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for 30 min. The resultant solution was filtered. Light-yellow prismatic single crystals of the title compound, suitable for *X*-ray structure determination, were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

Refinement

The OH and C-bound H atoms were included in calculated positions and treated as riding atoms: O-H = 0.82 Å, C-H = 0.93 and 0.96 Å, with $U_{iso}(H) = k \times U_{eq}(O,C)$ where k = 1.5 for OH and CH_3 H atoms and = 1.2 for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

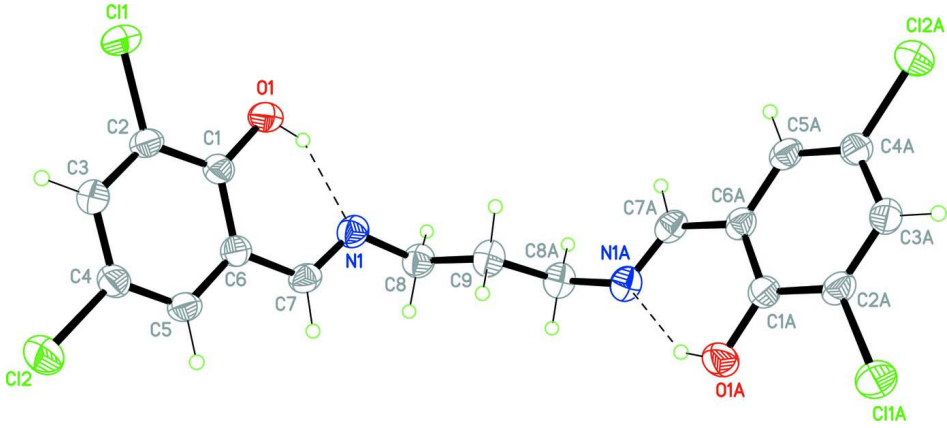

Figure 1

The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering [symmetry code for suffix A = -x, -y, z].

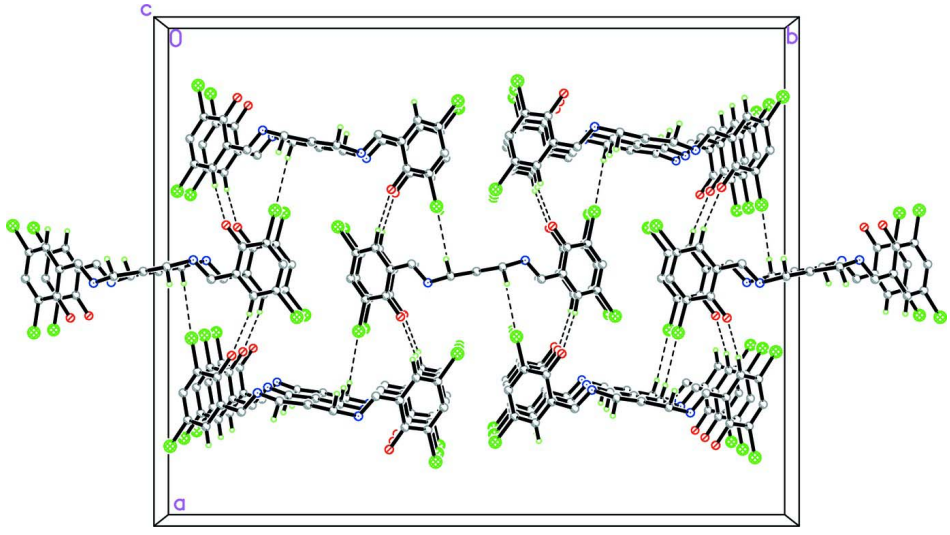

Figure 2

The crystal packing diagram of the title compound viewed down the *c*-axis, showing linking of molecules through C—H···O and weak C—H···Cl interactions (dashed lines).

4,4′,6,6′-Tetrachloro-2,2′-[(1*E*,1′*E*)-propane-1,3- diylbis(nitrilomethanylylidene)]diphenol

Crystal data

$C_{17}H_{14}Cl_4N_2O_2$
$M_r = 420.10$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 24.9797\ (14)$ Å
$b = 31.666\ (3)$ Å
$c = 4.4495\ (2)$ Å
$V = 3519.6\ (4)$ Å^3
$Z = 8$
$F(000) = 1712$
$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1234 reflections
$\theta = 2.5–27.5°$
$\mu = 0.69$ mm^{-1}
$T = 291$ K
Prism, light-yellow
0.26 × 0.23 × 0.18 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.842, T_{max} = 0.886
7926 measured reflections
1960 independent reflections
1634 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.3°, θ_{min} = 2.1°
h = −32→32
k = −40→40
l = −5→5

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.03
1960 reflections
115 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 1.6825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 842 Friedel pairs
Flack parameter: 0.08 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	−0.04810 (7)	0.14212 (6)	0.7828 (4)	0.0378 (4)	
C2	−0.05597 (7)	0.17577 (6)	0.5854 (5)	0.0405 (5)	
C3	−0.01420 (7)	0.19985 (6)	0.4810 (5)	0.0429 (5)	
H9	−0.0203	0.2218	0.3466	0.052*	
C4	0.03720 (8)	0.19078 (6)	0.5799 (5)	0.0419 (5)	
C5	0.04673 (7)	0.15902 (6)	0.7793 (5)	0.0409 (5)	
H2	0.0814	0.1538	0.8453	0.049*	
C6	0.00437 (7)	0.13440 (6)	0.8845 (4)	0.0374 (4)	
C7	0.01466 (8)	0.10036 (6)	1.0958 (4)	0.0401 (5)	
H4	0.0489	0.0972	1.1753	0.048*	
C8	−0.00878 (9)	0.03959 (6)	1.3730 (4)	0.0458 (5)	
H5A	−0.0378	0.0351	1.5143	0.055*	
H5B	0.0234	0.0461	1.4863	0.055*	
C9	0.0000	0.0000	1.1876 (7)	0.0465 (7)	
H6A	−0.0309	−0.0043	1.0589	0.056*	0.50

H6B	0.0309	0.0043	1.0589	0.056*	0.50
Cl1	−0.12035 (2)	0.187207 (19)	0.46609 (15)	0.05937 (17)	
Cl2	0.09006 (2)	0.220654 (18)	0.43888 (16)	0.06188 (18)	
N1	−0.02186 (7)	0.07479 (5)	1.1743 (4)	0.0424 (4)	
O1	−0.08951 (5)	0.11861 (4)	0.8692 (4)	0.0503 (4)	
H1	−0.0790	0.0999	0.9826	0.075*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0349 (9)	0.0355 (9)	0.0429 (12)	−0.0002 (8)	−0.0034 (8)	−0.0052 (9)
C2	0.0322 (9)	0.0401 (10)	0.0490 (12)	0.0056 (8)	−0.0083 (8)	−0.0044 (9)
C3	0.0415 (10)	0.0356 (9)	0.0518 (13)	0.0015 (8)	−0.0029 (10)	0.0001 (10)
C4	0.0349 (10)	0.0359 (10)	0.0550 (13)	−0.0016 (8)	0.0015 (9)	−0.0067 (9)
C5	0.0314 (9)	0.0402 (10)	0.0512 (13)	0.0052 (8)	−0.0060 (8)	−0.0087 (9)
C6	0.0368 (9)	0.0344 (9)	0.0409 (11)	0.0028 (7)	−0.0035 (8)	−0.0085 (9)
C7	0.0374 (10)	0.0420 (11)	0.0410 (11)	0.0067 (8)	−0.0083 (8)	−0.0084 (9)
C8	0.0521 (12)	0.0446 (11)	0.0407 (12)	0.0032 (9)	−0.0053 (9)	−0.0001 (10)
C9	0.0567 (17)	0.0430 (15)	0.0399 (15)	0.0048 (13)	0.000	0.000
Cl1	0.0371 (3)	0.0610 (3)	0.0800 (4)	0.0024 (2)	−0.0162 (3)	0.0145 (3)
Cl2	0.0434 (3)	0.0532 (3)	0.0891 (5)	−0.0076 (2)	0.0080 (3)	0.0055 (3)
N1	0.0464 (9)	0.0411 (9)	0.0397 (9)	0.0039 (7)	−0.0062 (8)	−0.0015 (8)
O1	0.0366 (7)	0.0503 (9)	0.0639 (10)	−0.0058 (6)	−0.0093 (6)	0.0124 (7)

Geometric parameters (Å, º)

C1—O1	1.331 (2)	C6—C7	1.453 (3)
C1—C2	1.395 (3)	C7—N1	1.269 (2)
C1—C6	1.408 (2)	C7—H4	0.9300
C2—C3	1.373 (3)	C8—N1	1.460 (3)
C2—Cl1	1.7318 (19)	C8—C9	1.517 (3)
C3—C4	1.387 (3)	C8—H5A	0.9700
C3—H9	0.9300	C8—H5B	0.9700
C4—C5	1.362 (3)	C9—C8^{i}	1.517 (3)
C4—Cl2	1.741 (2)	C9—H6A	0.9700
C5—C6	1.395 (3)	C9—H6B	0.9700
C5—H2	0.9300	O1—H1	0.8200
O1—C1—C2	119.98 (16)	N1—C7—C6	121.61 (17)
O1—C1—C6	122.23 (17)	N1—C7—H4	119.2
C2—C1—C6	117.79 (17)	C6—C7—H4	119.2
C3—C2—C1	122.02 (17)	N1—C8—C9	109.52 (18)
C3—C2—Cl1	119.06 (15)	N1—C8—H5A	109.8
C1—C2—Cl1	118.92 (15)	C9—C8—H5A	109.8
C2—C3—C4	118.75 (19)	N1—C8—H5B	109.8
C2—C3—H9	120.6	C9—C8—H5B	109.8
C4—C3—H9	120.6	H5A—C8—H5B	108.2
C5—C4—C3	121.42 (18)	C8^{i}—C9—C8	114.1 (3)
C5—C4—Cl2	120.22 (15)	C8^{i}—C9—H6A	108.7
C3—C4—Cl2	118.35 (16)	C8—C9—H6A	108.7

C4—C5—C6	119.90 (17)	C8^{i}—C9—H6B	108.7
C4—C5—H2	120.1	C8—C9—H6B	108.7
C6—C5—H2	120.1	H6A—C9—H6B	107.6
C5—C6—C1	120.07 (18)	C7—N1—C8	119.53 (17)
C5—C6—C7	119.82 (17)	C1—O1—H1	109.5
C1—C6—C7	120.10 (18)		
O1—C1—C2—C3	−177.41 (19)	C4—C5—C6—C7	179.60 (17)
C6—C1—C2—C3	2.7 (3)	O1—C1—C6—C5	177.77 (18)
O1—C1—C2—Cl1	2.0 (2)	C2—C1—C6—C5	−2.4 (3)
C6—C1—C2—Cl1	−177.84 (15)	O1—C1—C6—C7	−1.3 (3)
C1—C2—C3—C4	−1.2 (3)	C2—C1—C6—C7	178.59 (16)
Cl1—C2—C3—C4	179.35 (16)	C5—C6—C7—N1	−173.18 (19)
C2—C3—C4—C5	−0.7 (3)	C1—C6—C7—N1	5.9 (3)
C2—C3—C4—Cl2	178.41 (15)	N1—C8—C9—C8^{i}	−174.57 (19)
C3—C4—C5—C6	1.0 (3)	C6—C7—N1—C8	176.32 (17)
Cl2—C4—C5—C6	−178.09 (15)	C9—C8—N1—C7	−97.64 (19)
C4—C5—C6—C1	0.5 (3)		

Symmetry code: (i) −*x*, −*y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.84	2.574 (2)	147
C5—H2···O1^{ii}	0.93	2.43	3.336 (2)	166
C8—H5*B*···Cl1^{iii}	0.97	2.89	3.851 (2)	169

Symmetry codes: (ii) *x*+1/4, −*y*+1/4, *z*+1/4; (iii) *x*+1/4, −*y*+1/4, *z*+5/4.